

# **SAMPLING QA/QC WORK PLAN**

**Central Steel and Drum  
NEWARK, ESSEX COUNTY, NEW JERSEY**

Prepared by

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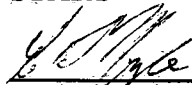
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Region II - Removal Action Branch  
Edison, New Jersey 08837**

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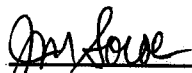
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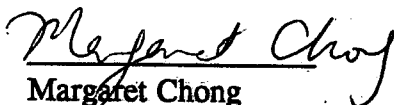
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320393



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## **1.0 BACKGROUND**

The Central Steel and Drum Site is an inactive and abandoned drum recycling and processing facility located at 704 Doremus Avenue in Newark, N.J. The facility consists of several buildings on 8.5 acres. The Site recycled used drums by cleaning, sandblasting of metals, incineration of residual drum contents, and repainting of restored drums for reuse. START has not had any previous work history at this site however the NJ DEP referral defines 1000 tires, 200 drums that may or may not have contents and an oil slick on nearby water surfaces as possible areas for further investigation (Note that the site is near a petroleum refinery and would therefore not represent EPA regulated contaminants.) The DEP site history notes high levels of volatile compounds, heavy metals, and chlordane from previous analysis during the years of operation until 1985. No recent DEP site information is listed since Nov 1994. The well documented site history and the established analytical results defined in the past will be used to confirm continuing conditions of contamination of the soils and ground water to the high levels of heavy metals at the site. The unknown current site conditions may not demonstrate continuing contamination with Field Characterization testing (Hazcatting) of organic wastes as normally done during assessments but will be included as an additional form of site characterization if conditions permit.

## **2.0 DATA USE OBJECTIVES**

The objective of this sampling event is to select sample locations for "in situ" XRF Lead analysis that may demonstrate the previously reported contamination levels found at the site.

## **3.0 QUALITY ASSURANCE OBJECTIVES**

The overall Quality Assurance (QA) Objective is to identify contaminants on site and to determine the type of contaminants using the SPECTRACE 9000 Field Portable X-Ray Fluorescence Instrument and the HAZCAT field compatibility kit.

The U.S. Environmental Protection Agency (EPA) On-Scene Coordinator (OSC) has specified a Level 1 QA Objective (QA-1). Details of this Assurance Level are provided in Section 6.0.

The QA-1 objective may be applied to these site specific activities:

1. Physical and/or Chemical properties of samples;
2. Waste Compatibility;
3. Hazard Characterization.

There will be no QA data collected for this objective.

The objective of this project/event applies to the following parameters:

<b>TABLE 1</b> <b>Quality Assurance Objectives</b>			
QA Parameters	Matrix	Intended use of data	QA Objective
Field Compatibility	Drum Liquids and/or Solids (if contaminants are found) Contaminated Soils and Ash	Hazard Characterization	QA-1

<b>TABLE 2</b> <b>QA/QC Analysis and Objectives Summary</b>				
MATRIX	ANALYTICAL PARAMETER	CONTAINER (SAMPLE) VOLUME	Analytical Method Reference	QA/QC Quantitation Limits
Various	Field Compatibility	One 4-oz. wide- mouth glass jar	HAZCAT Manual & XRF SOP 1713	N/A

#### **4.0 APPROACH AND SAMPLING METHODOLOGIES**

As stated above, START will perform the following tasks:

1. Drum ,Soil and Water inspection/visual observations and logging information;
2. Field compatibility tests.

##### **4.1 Sampling Equipment**

XRF Sampling locations will be identified by the OSC for Lead "in situ" Analysis. EPA/ERT SOP #2009, Drum Sampling, will be used if drummed waste is found.

##### **4.2 Sampling Design**

It is anticipated that no more than fifteen (15) sample locations will be tested. The drums of waste if found to be present to be sampled will be chosen by the OSC.

### **4.3 Standard Operating Procedures (SOPs)**

#### **4.3.1 Sample Documentation**

All sample documents will be legibly completed using waterproof ink. Any corrections or revisions will be made by lining once through the incorrect entry and initialing the error.

#### **FIELD LOGBOOK**

The field logbook is essentially a descriptive notebook detailing site activities and observations so that an accurate account of field procedures can be reconstructed in the writer's absence. Logbook entries should record (at a minimum) the following:

1. Site name, code, and project number;
2. Project start and end dates;
3. Dates and times of all entries (military time) and the name of the individual making the entry;
4. Name(s) of personnel on site and sampling team members;
5. Descriptions of all site activities, including site and exclusion zone entry and exit times, and level of PPE used by team members;
6. Noteworthy events and discussions;
7. Weather conditions;
8. Site observations;
9. Identification and detailed description of each sample and station location including: the sample type (grab or composite); preservation; sampling depth (if applicable); type of sampling equipment used; compass and distance measurements (as applicable); and the name of the sampling team member.
10. Identification number(s) and calibration date(s) of portable monitoring and/or field analytical equipment used to obtain measurements;

11. Subcontractor information and names of on-site personnel;
12. Date and time of sample collections, along with chain of custody and air courier service information;
13. Record of photographs; and
14. Sketches, including site layout and sample station locations.

#### **DRUM INVENTORY LOG**

See Attachment B for a copy of the drum inventory log sheet to be used for this project.

#### **4.3.2 Sampling SOPs**

#### **DRUM SAMPLING**

Drum sampling activities will be conducted in accordance with guidelines outlined in EPA/ERT Drum Sampling SOP #2009 (Attachment C).

#### **4.3.3 Sample Handling and Shipment**

All field testing will be done on site. Samples will not be collected for additional analysis.

#### **4.4 Analytical Methods/Test Procedures**

All field testing for RCRA characteristics and additional parameters will be performed using a HAZCAT kit. All testing will be conducted in accordance with the HazCat field directional manual.

#### **4.5 Schedule of Activities**

TABLE 3 PROPOSED SCHEDULE OF WORK		
Start Date	Activity	End Date
14 May 1997	Field Compatibility	14 May 1997

#### **4.6 Disposal of PPE and contaminated sampling materials**

Because the site is not secure, all PPE and sampling material will be placed in plastic garbage bags and transported from the site.



## 5.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The OSC, Margaret Chong, will provide overall direction to the staff concerning project sampling needs, objectives and schedule. The START PM, Ed Moyle, will be the primary point of contact with the OSC. The START PM is responsible for the development and completion of the Sampling QA/QC Plan, project team organization, and supervision of all project tasks, including reporting and deliverables. The START PM will act as the Site QC Coordinator and will be responsible for ensuring field adherence to the Sampling QA/QC Plan and recording of any deviations.

The following personnel will conduct work on the Assessment and Removal phases of this project:

<u>Personnel</u>	<u>Affiliation</u>	<u>Responsibility</u>
Ed Moyle	Region II START	Project Manager/QAQC
Brian McGinn	Region II START	Sampler
Michael Mahnkoph	Region II START	Sampler
Christoph Stannik	Region II START	Field Screening
Donielle Perri	Region II START	XRF Analysis
Joesph Soroka	Region II START	XRF Analysis
Tom Oneal (1/2 day)	Region II START	Consultant (as a former DEP inspector at this site when it was in operation)

## 6.0 QA REQUIREMENTS

The following QA Protocols for a Level 1 QA data are applicable to all sample matrices and include:

1. Documentation in the form of field logbooks, appropriate field data sheets and appropriate drum log inventory sheets:
2. Performance check of the appropriate test method (i.e. test strips) will be summarized and documented in the field logbook.

## 7.0 DELIVERABLES

Drum inventory logs will be filled out for every sample taken. A copy of the logs will be provided to the OSC. A Trip Report will be prepared to provide a detailed accounting of what occurred during the sampling event.

## **Maps/Figures**

A map depicting the site layout and areas will be included in the Trip Report as appropriate.

## **8.0 DATA VALIDATION**

### **QA Level 1**

No data validation is required under QA level 1

## **9.0 SYSTEM AUDIT**

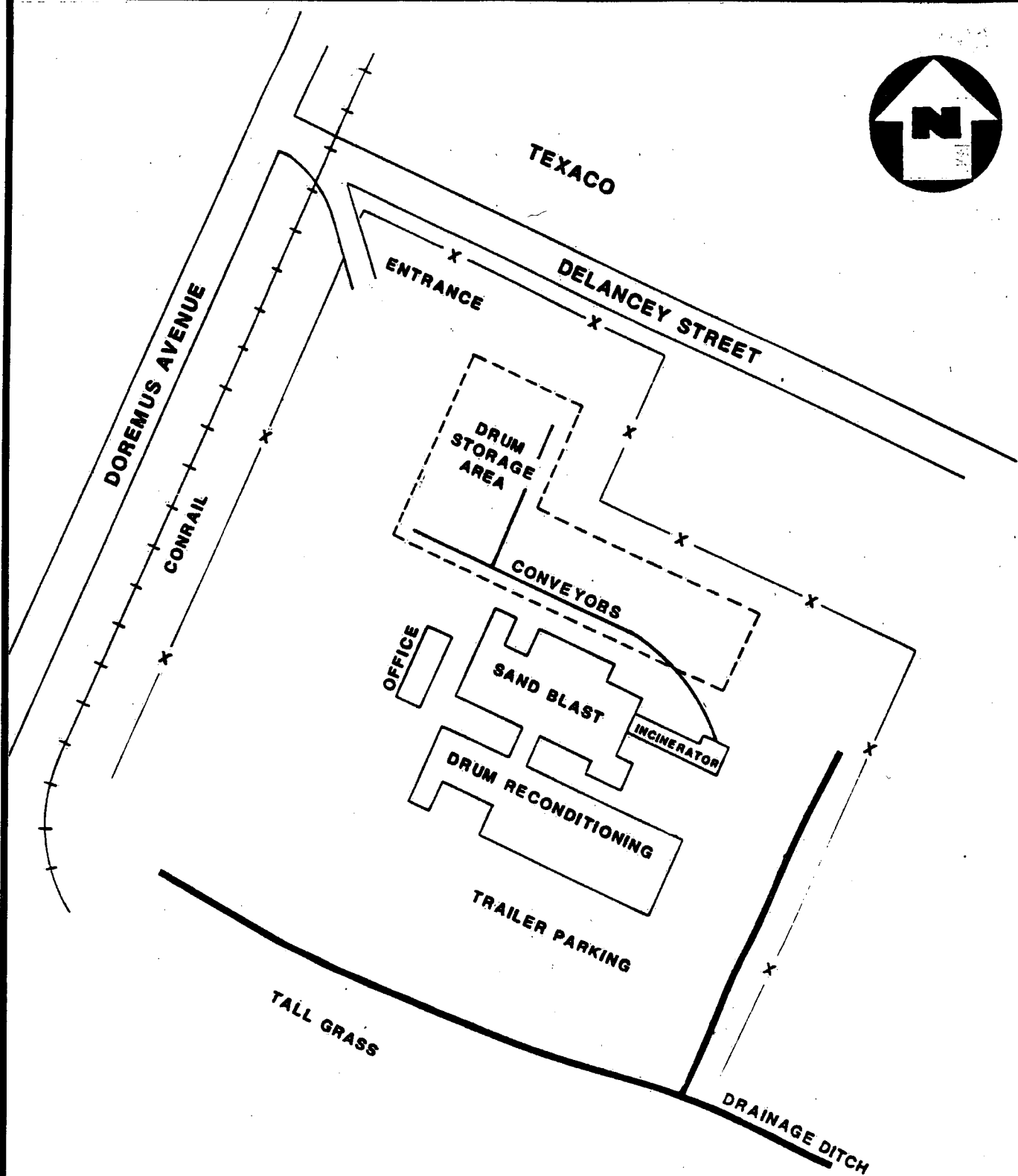
The field QA/QC officer will observe sampling and hazcating operations to ensure compliance with the QA/QC requirements.

## **10.0 CORRECTIVE ACTION**

All provisions will be taken in the field to ensure that any problems that may develop will be dealt with as quickly as possible to ensure the continuity of the project/sampling event. Any deviations from this plan will be noted in the site/field log book.

**ATTACHMENT A**

**SITE MAP**



Source: Halliburton NUS

(NOT TO SCALE)



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EPA PM

M. Chong

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START PM

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Figure 2:  
Site Layout and  
Sampling Locations

**ATTACHMENT B**  
**DRUM INVENTORY LOG**

SITE NAME: \_\_\_\_\_ SAMPLE NO: \_\_\_\_\_ DRUM NUMBER: \_\_\_\_\_

GRID LOCATION FOUND: \_\_\_\_\_ STAGING LOCATION: \_\_\_\_\_

LOGGER: \_\_\_\_\_ SAMPLER: \_\_\_\_\_

PROJECT NO: \_\_\_\_\_ DATE/TIME: \_\_\_\_\_

## DRUM DESCRIPTION:

CONSTRUCTION		TYPE		CONDITION:		
Fiber <input type="checkbox"/>	Poly <input type="checkbox"/>	Poly Lined <input type="checkbox"/>	Overpack <input type="checkbox"/>	rusted <input type="checkbox"/>	leaking <input type="checkbox"/>	damaged <input type="checkbox"/>
Steel <input type="checkbox"/>	Nickel <input type="checkbox"/>	Open Top <input type="checkbox"/>	Ring Top <input type="checkbox"/>	bulging <input type="checkbox"/>	perforated <input type="checkbox"/>	good <input type="checkbox"/>
Stainless Steel <input type="checkbox"/>	Other <input type="checkbox"/>	Closed Top <input type="checkbox"/>	other _____			

DRUM SIZE (Gallons): 85 ☐ 55 ☐ 42 ☐ 30 ☐ 15 ☐ 10 ☐ 5 ☐ Other \_\_\_\_\_

MFG NAME \_\_\_\_\_

CHEMICAL NAME \_\_\_\_\_

DRUM MARKINGS \_\_\_\_\_

DRUM LABELS \_\_\_\_\_

FIELD AIR MONITORING INSTRUMENT READINGS: HNa \_\_\_\_\_ OVA \_\_\_\_\_ CGI \_\_\_\_\_ RAD METER \_\_\_\_\_ OTHER \_\_\_\_\_

## PHYSICAL DESCRIPTION:

Layers			Physical			Color/Description <sup>1</sup>	Clarity			Solubility		Reaction	
P H A S E	I N C H E S	L I Q U I D	S O L I D	S L U D G E	G E L	<sup>1</sup> - Oil, Syrup, Viscuous. Watery, Paste, Chunks, Gel, Spongy, Soaplike. Soft, Hard Powder Crystal Granular, Rubbery	C L E A R	C L O U D Y	O P A Q U E	W A T E R	H E X A N E	A I R	W A T E R
Top													
Middle													
Bottom													

## HAZCAT RESULTS:

Layers	pH	Chlorine not wire	Flammable	Cyanide	Oxidizer	Chloride	Peroxide	Mercury	Sulfide	PCB
Top										
Middle										
Bottom										

## ASSIGNED WASTE STREAM - BASED ON INITIAL RCRA HAZARD

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## TEST COMPATABILITY RESULTS:

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**ATTACHMENT C**  
**DRUM SAMPLING SOP #2009**

## 2.0 DRUM SAMPLING: SOP #2009

### 2.1 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide technical guidance on safe and cost-effective response actions at hazardous waste sites containing drums with unknown contents. Container contents are sampled and characterized for disposal, bulking, recycling, grouping, and/or classification purposes.

### 2.2 METHOD SUMMARY

Prior to sampling, drums must be inventoried, staged, and opened. An inventory entails recording visual qualities of each drum and any characteristics pertinent to the contents' classification. Staging involves the organization, and sometimes consolidation of drums which have similar wastes or characteristics. Opening of closed drums can be performed manually or remotely. Remote drum opening is recommended for worker safety. The most widely used method of sampling a drum involves the use of a glass thief. This method is quick, simple, relatively inexpensive, and requires no decontamination.

### 2.3 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples collected from drums are considered waste samples. No preservatives should be added since there is a potential reaction of the sample with the preservative. Samples should, however, be cooled to 4°C and protected from sunlight in order to minimize any potential reaction due to the light sensitivity of the sample.

Sample bottles for collection of waste liquids, sludges, or solids are typically wide-mouth amber jars with Teflon-lined screw caps. Actual volume required for analysis should be determined in conjunction with the laboratory performing the analysis.

Follow these waste sample handling procedures:

1. Place sample container in two Ziploc plastic bags.

2. Place each bagged container in a 1-gallon covered can containing absorbent packing material. Place the lid on the can.
3. Mark the sample identification number on the outside of the can.
4. Place the marked cans in a cooler, and fill remaining space with absorbent packing material.
5. Fill out chain of custody form for each cooler, place in plastic, and affix to inside lid of cooler.
6. Secure and custody seal the lid of cooler.
7. Arrange for the appropriate transportation mode consistent with the type of hazardous waste involved.

### 2.4 INTERFERENCES AND POTENTIAL PROBLEMS

The practice of tapping drums to determine their contents is neither safe nor effective and should not be used if the drums are visually overpressurized or if shock-sensitive materials are suspected. A laser thermometer may be used instead.

Drums that have been overpressurized, to the extent that the head is swollen several inches above the level of the chime, should not be moved. A number of devices have been developed for venting critically swollen drums. One method that has proven to be effective is a tube and spear device. A light aluminum tube (3 meters long) is positioned at the vapor space of the drum. A rigid, hooking device attached to the tube goes over the chime and holds the tube securely in place. The spear is inserted in the tube and positioned against the drum wall. A sharp blow on the end of the spear drives the sharpened tip through the drum and the gas vents along the grooves. The venting should be done from behind a wall or barricade. This device can be cheaply and easily designed and constructed where needed. Once the pressure has been relieved, the bung can be removed and the drum sampled.



## 2.5 EQUIPMENT/APPARATUS

The following are standard materials and equipment required for sampling:

- personal protection equipment
- wide-mouth glass jars with Teflon cap liner, approximately 500 mL volume
- uniquely numbered sample identification labels with corresponding data sheets
- 1-gallon covered cans half-filled with absorbent (vermiculite)
- chain of custody forms
- decontamination materials
- glass thief tubes or Composite Liquid Waste Samplers (COLIWASA)
- laser thermometer
- drum opening devices

Drum opening devices include the following:

### 2.5.1 Bung Wrench

A common method for opening drums manually is using a universal bung wrench. These wrenches have fittings made to remove nearly all commonly encountered bungs. They are usually constructed of cast iron, brass, or a bronze-beryllium, non-sparking alloy formulated to reduce the likelihood of sparks. The use of a non-sparking bung wrench does not completely eliminate the possibility of a spark being produced. (See Figure 1, Appendix B.)

### 2.5.2 Drum Deheader

When a bung is not removable with a bung wrench, a drum can be opened manually by using a drum deheader. This tool is constructed of forged steel with an alloy steel blade and is designed to cut the lid of a drum off or part way off by means of a scissors-like cutting action. A limitation of this device is that it can be attached only to closed head drums. Drums with removable heads must be opened by other means. (See Figure 2, Appendix B.)

### 2.5.3 Hand Pick, Pickaxe, and Hand Spike

These tools are usually constructed of brass or a non-sparking alloy with a sharpened point that can penetrate the drum lid or head when the tool is swung. The hand picks or pickaxes that are most

commonly used are commercially available; whereas the spikes are generally uniquely fabricated 4-foot long poles with a pointed end. (See Figure 3, Appendix B.)

### 2.5.4 Backhoe Spike

The most common means used to open drums remotely for sampling is the use of a metal spike attached or welded to a backhoe bucket. In addition to being very efficient, this method can greatly reduce the likelihood of personal exposure. (See Figure 4, Appendix B.)

### 2.5.5 Hydraulic Drum Opener

Another remote method for opening drums is with remotely operated hydraulic devices. One such device uses hydraulic pressure to pierce through the wall of a drum. It consists of a manually operated pump which pressurizes soil through a length of hydraulic line. (See Figure 5, Appendix B.)

### 2.5.6 Pneumatic Devices

A pneumatic bung remover consists of a compressed air supply that is controlled by a heavy-duty, two-stage regulator. A high-pressure air line of desired length delivers compressed air to a pneumatic drill, which is adapted to turn a bung fitting selected to fit the bung to be removed. An adjustable bracketing system has been designed to position and align the pneumatic drill over the bung. This bracketing system must be attached to the drum before the drill can be operated. Once the bung has been loosened, the bracketing system must be removed before the drum can be sampled. This remote bung opener does not permit the slow venting of the container, and therefore appropriate precautions must be taken. It also requires the container to be upright and relatively level. Bungs that are rusted shut cannot be removed with this device. (See Figure 6, Appendix B.)

## 2.6 REAGENTS

Reagents are not typically required for preserving drum samples. However, reagents are used for decontaminating sampling equipment. Decontamination solutions are specified in ERT SOP #2006, Sampling Equipment Decontamination.

## 2.7 PROCEDURES

### 2.7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are needed.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or preclean equipment, and ensure that it is in working order.
4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.
6. Use stakes, flagging, or buoys to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

### 2.7.2 Drum Inspection

Appropriate procedures for handling drums depend on the contents. Thus, prior to any handling, drums should be visually inspected to gain as much information as possible about their contents. Those in charge of inspections should be on the look-out for:

- drum condition, corrosion, rust, and leaking contents
- symbols, words, or other markings on the drum indicating hazards (i.e., explosive, radioactive, toxic, flammable)
- signs that the drum is under pressure
- shock sensitivity

Monitor around the drums with radiation instruments, organic vapor monitors (OVA) and combustible gas indicators (CGI).

Classify the drums into categories, for instance:

- radioactive
- leaking/deteriorating
- bulging
- drums containing lab packs
- explosive/shock sensitive

All personnel should assume that unmarked drums contain hazardous materials until their contents have been categorized, and that labels on drums may not accurately describe their contents.

If it is presumed that there are buried drums on-site, geophysical investigation techniques such as magnetometry, ground penetrating radar, and metal detection can be employed in an attempt to determine depth and location of the drums. See ERT SOP #2159, General Surface Geophysics.

### 2.7.3 Drum Staging

Prior to sampling, the drums should be staged to allow easy access. Ideally, the staging area should be located just far enough from the drum opening area to prevent a chain reaction if one drum should explode or catch fire when opened.

While staging, physically separate the drums into the following categories: those containing liquids, those containing solids, lab packs, or gas cylinders, and those which are empty. This is done because the strategy for sampling and handling drums/containers in each of these categories will be different. This may be achieved by:

- Visual inspection of the drum and its labels, codes, etc. Solids and sludges are typically disposed of in open-top drums. Closed-head drums with a bung opening generally contain liquid.
- Visual inspection of the contents of the drum during sampling followed by restaging, if needed.

Once a drum has been excavated and any immediate hazard has been eliminated by overpacking or transferring the drum's contents, affix a numbered tag to the drum and transfer it to a staging area. Color-coded tags, labels, or bands should be used to mark similar waste types. Record a description of each drum, its condition, any unusual markings, and the location where it was buried or stored, on a drum data sheet (Appendix A). This data sheet becomes the principal

recordkeeping tool for tracking the drum onsite.

Where there is good reason to suspect that some drums contain radioactive, explosive, and shock-sensitive materials, these drums should be staged in a separate, isolated area. Placement of explosives and shock-sensitive materials in diked and fenced areas will minimize the hazard and the adverse effects of any premature detonation of explosives.

Where space allows, the drum opening area should be physically separated from the drum removal and drum staging operations. Drums are moved from the staging area to the drum opening area one at a time using forklift trucks equipped with drum grabbers or a barrel grappler. In a large-scale drum handling operation, drums may be conveyed to the drum opening area using a roller conveyor.

## 2.7.4 Drum Opening

There are three basic techniques available for opening drums at hazardous waste sites:

- Manual opening with non-sparking bung wrenches,
- Drum deheading, and
- Remote drum puncturing or bung removal.

The choice of drum opening techniques and accessories depends on the number of drums to be opened, their waste contents, and physical condition. Remote drum opening equipment should always be considered in order to protect worker safety. Under OSHA 1910.120, manual drum opening with bung wrenches or deheaders should be performed only with structurally sound drums having contents that are known to be (1) not shock sensitive, (2) non-reactive, (3) non-explosive, and (4) non-flammable.

### *Manual Drum Opening with a Bung Wrench*

Manual drum opening with bung wrenches (Figure 1, Appendix B) should not be performed unless the drums are structurally sound (no evidence of bulging or deformation) and their contents are known to be non-explosive. If opening the drum with bung wrenches is deemed reasonably cost-effective and safe, then follow these procedures to minimize the hazard:

1. Fully outfit field personnel with protective gear.
2. Position drum upright with the bung up, or, for drums with bungs on the side, lay the drum on its side with the bung plug up.
3. Wrench the bung with a slow, steady pulling motion across the drum. If the length of the bung wrench handle provides inadequate leverage for unscrewing the plug, attach a "cheater bar" to the handle to improve leverage.

### *Manual Drum Opening with a Drum Deheader*

Drums are opened with a drum deheader (Figure 2, Appendix B) by first positioning the cutting edge just inside the top chime and then tightening the adjustment screw so that the deheader is held against the side of the drum. Moving the handle of the deheader up and down while sliding the deheader along the chime will cut off the entire top. If the top chime of a drum has been damaged or badly dented, it may not be possible to cut off the entire top. Since there is always the possibility that a drum may be under pressure, make the initial cut very slowly to allow for the gradual release of any built-up pressure. A safer technique would be to use a remote method to puncture the drum prior to using the deheader.

Self-propelled drum openers which are either electrically or pneumatically driven can be used for quicker and more efficient deheading.

### *Manual Drum Opening with a Hand Pick, Pickaxe, or Spike*

When a drum must be opened and neither a bung wrench nor a drum deheader is suitable, the drum can be opened for sampling by using a hand pick, pickaxe, or spike (Figure 3, Appendix B). Often the drum lid or head must be hit with a great deal of force in order to penetrate it. The potential for splash or spraying is greater than with other opening methods and, therefore, this method of drum opening is not recommended, particularly when opening drums containing liquids. Some spikes used have been modified by the addition of a circular splash plate near the penetrating end. This plate acts as a shield and reduces the amount of splash in the direction of the person using the spike. Even with this shield, good splash gear is essential.

Since drums cannot be opened slowly with these tools, spray from drums is common requiring appropriate safety measures. Decontaminate the pick or spike after each drum is opened to avoid cross-contamination and/or adverse chemical reaction from incompatible materials.

### *Remote Drum Opening with a Backhoe Spike*

Remotely operated drum opening tools are the safest available means of drum opening. Remote drum opening is slow, but is much safer compared to manual methods of opening.

Drums should be "staged" or placed in rows with adequate aisle space to allow ease in backhoe maneuvering. Once staged, the drums can be quickly opened by punching a hole in the drum head or lid with the spike.

The spike (Figure 4, Appendix B) should be decontaminated after each drum is opened to prevent cross-contamination. Even though some splash or spray may occur when this method is used, the operator of the backhoe can be protected by mounting a large shatter-resistant shield in front of the operator's cage. This, combined with the required level of personal protection gear, should be sufficient to protect the operator. Additional respiratory protection can be afforded by providing the operator with an on-board airline system.

### *Remote Drum Opening with Hydraulic Devices*

A piercing device with a metal point is attached to the end of a hydraulic line and is pushed into the drum by hydraulic pressure (Figure 5, Appendix B). The piercing device can be attached so that the sampling hole can be made on either the side or the head of the drum. Some of the metal piercers are hollow or tube-like so that they can be left in place if desired and serve as a permanent tap or sampling port. The piercer is designed to establish a tight seal after penetrating the container.

### *Remote Drum Opening with Pneumatic Devices*

Pneumatically-operated devices utilizing compressed air have been designed to remove drum bungs remotely (Figure 6, Appendix B).

## **2.7.5 Drum Sampling**

After the drum has been opened, monitor headspace gases using an explosimeter and organic vapor analyzer. In most cases it is impossible to observe the contents of these sealed or partially sealed vessels. Since some layering or stratification is likely in any solution left undisturbed over time, take a sample that represents the entire depth of the vessel.

When sampling a previously sealed vessel, check for the presence of a bottom sludge. This is easily accomplished by measuring the depth to the apparent bottom, then comparing it to the known interior depth.

### *Glass Thief Sampler*

The most widely used implement for sampling is a glass tube commonly referred to as a glass thief (Figure 7, Appendix B). This tool is simple, cost effective, quick, and collects a sample without having to decontaminate. Glass thieves are typically 6mm to 16mm I.D. and 48 inches long.

Procedures for using a glass thief are as follows:

1. Remove cover from sample container.
2. Insert glass tubing almost to the bottom of the drum or until a solid layer is encountered. About one foot of tubing should extend above the drum.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Cap the top of the sampling tube with a tapered stopper or thumb, ensuring liquid does not come into contact with stopper.
5. Carefully remove the capped tube from the drum and insert the uncapped end in the sample container.
6. Release stopper and allow the glass thief to drain until the container is approximately 2/3 full.
7. Remove tube from the sample container, break it into pieces and place the pieces in the drum.

8. Cap the sample container tightly and place prelabeled sample container in a carrier.
9. Replace the bung or place plastic over the drum.
10. Log all samples in the site logbook and on field data sheets.
11. Package samples and complete necessary paperwork.
12. Transport sample to decontamination zone to prepare it for transport to the analytical laboratory.

In many instances a drum containing waste material will have a sludge layer on the bottom. Slow insertion of the sample tube down into this layer and then a gradual withdrawal will allow the sludge to act as a bottom plug to maintain the fluid in the tube. The plug can be gently removed and placed into the sample container by the use of a stainless steel lab spoon.

It should be noted that in some instances disposal of the tube by breaking it into the drum may interfere with eventual plans for the removal of its contents. This practice should be cleared with the project officer or other disposal techniques evaluated.

### *COLIWASA Sampler*

Some equipment is designed to collect a sample from the full depth of a drum and maintain it in the transfer tube until delivery to the sample bottle. These designs include primarily the Composite Liquid Waste Sampler (COLIWASA) and modifications thereof. The COLIWASA (Figure 8, Appendix B) is a much cited sampler designed to permit representative sampling of multiphase wastes from drums and other containerized wastes. One configuration consists of a 152 cm by 4 cm I.D. section of tubing with a neoprene stopper at one end attached by a rod running the length of the tube to a locking mechanism at the other end.

Manipulation of the locking mechanism opens and closes the sampler by raising and lowering the neoprene stopper. One model of the COLIWASA is shown in Appendix B; however, the design can be modified and/or adapted somewhat to meet the needs of the sampler.

The major drawbacks associated with using a COLIWASA concern decontamination and costs. The sampler is difficult, if not impossible to decontaminate in the field and its high cost in relation to alternative procedures (glass tubes) make it an impractical throwaway item. It still has applications, however, especially in instances where a true representation of a multiphase waste is absolutely necessary.

Follow these procedures for using the COLIWASA:

1. Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.
2. Slowly lower the sampler into the liquid waste. Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sample tube is lower than that outside the sampler, the sampling rate is too fast and will result in a non-representative sample.
3. When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block.
4. Slowly withdraw the sample from the waste container with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.
5. Carefully discharge the sample into a suitable sample container by slowly pulling the lower end of the T-handle away from the locking block while the lower end of the sampler is positioned in a sample container.
6. Cap the sample container tightly and place prelabeled sample container in a carrier.
7. Replace the bung or place plastic over the drum.
8. Log all samples in the site logbook and on field data sheets.

9. Package samples and complete necessary paperwork.
10. Transport sample to decontamination zone to prepare it for transport to the analytical laboratory.

## **2.8 CALCULATIONS**

This section is not applicable to this SOP.

## **2.9 QUALITY ASSURANCE/ QUALITY CONTROL**

The following general quality assurance procedures apply:

- Document all data on standard chain of custody forms, field data sheets, or within site logbooks.
- Operate all instrumentation in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

## **2.10 DATA VALIDATION**

This section is not applicable to this SOP.

## **2.11 HEALTH AND SAFETY**

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and specific health and safety procedures.

The opening of closed containers is one of the most hazardous site activities. Maximum efforts should be made to ensure the safety of the sampling team. Proper protective equipment and a general awareness of the possible dangers will minimize the risk inherent in sampling operations. Employing proper drum-opening techniques and equipment will also safeguard personnel. Use remote sampling equipment whenever feasible.

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RESPONSE ENGINEERING AND ANALYTICAL CONTRACT

STANDARD OPERATING PROCEDURES

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OPERATING PROCEDURE

U.S.EPA CONTRACT 68-C4-0022

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## 1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to serve as a guide to the start-up, check out, operation, calibration, and routine use of the Spectrace 9000 field portable x-ray fluorescence instrument for field use in screening hazardous or potentially hazardous inorganic materials. It is not intended to replace or diminish the use of the Spectrace 9000 Operating Instructions. The Operating Instructions contain additional information for optimizing instrument performance and for utilizing different applications.

The procedures contained herein are general operating guidelines which may be changed as required, depending on site conditions, equipment limitations, limitations imposed by Quality Assurance/Quality Control (QA/QC) procedure or other protocol limitations. In all instances, the procedures finally employed should be documented and included in any or all final reports. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

### 1.1 Principles of Operation

X-ray Fluorescence (XRF) spectroscopy is a non destructive qualitative and quantitative analytical technique used to determine the chemical composition of samples. In a source excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate samples. During interaction with samples, source X-rays may either undergo scattering (dominating process) or absorption by sample atoms in a process known as the photoelectric effect (absorption coefficient). This phenomenon originates when incident radiation knocks out an electron from the innermost shell of an atom creating a vacancy. The atom is excited and releases its surplus energy almost instantly by filling the vacancy with an electron from one of the higher energy shells. This rearrangement of electrons is associated with the emission of X-rays characteristic (in terms of energy) of the given atom. This process is referred to as emission of fluorescent X-rays (fluorescent yield). The overall efficiency of the fluorescence process is referred to as excitation efficiency and is proportional to the product of the absorption coefficient and the fluorescent yield.

#### 1.1.1 Characteristic X-rays

The Spectrace 9000 utilizes characteristic X-ray lines originating from the innermost shells of the atoms: K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for any element and, therefore, are the preferred analytical lines. The K lines are always accompanied by the L and M lines of the same element. However, with energies much lower than those of the K lines, they can usually be neglected for those elements for which the K lines are analytically useful. For heavy elements such as cerium (Ce) (atomic number [Z]=58), to uranium (U, Z=92), the L lines are the preferred lines for analysis. The  $L_{\alpha}$  and  $L_{\beta}$  lines have almost equal intensities, and the choice of one or the other depends on what interfering lines might be present. A source just energetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines.

The Spectrace 9000 Operating Instructions contain a table that identifies the X-rays (K or L) and elements measured for each excitation source.

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An X-ray source can excite characteristic X-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element (e.g., K absorption edge, L absorption edge, M absorption edge). The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies, and the L absorption edge energy is approximately the sum of the L and M line energies of the particular element.

Energies of the characteristic fluorescent X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer (electronic unit) measures the pulse amplitudes, which is the basis of a qualitative X-ray analysis. The number of counts at a given energy is representative of element concentration in a sample and is the basis for quantitative analysis.

#### 1.1.2 Scattered X-rays

The source radiation is scattered from the sample by two physical processes: coherent or elastic scattering (no energy loss), and Compton or inelastic scattering (small energy loss). Thus, source backscatter (background signal) actually consists of two components with X-ray lines close together. The higher energy line is equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. Furthermore, the scattered X-rays have the highest energies in the spectrum and, therefore, contribute most of the total measured intensity signal.

#### 1.2 Sample Types

Solid and liquid samples can be analyzed for elements aluminum (Al) through uranium (U) with proper X-ray source selection and instrument calibration. Typical environmental applications are:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings, etc.), sludges, and liquids (e.g., lead (Pb) in gasoline)
- Light elements in liquids (e.g., phosphorus [P], sulphur [S], and chlorine [Cl] in organic solutions)
- Heavy metals in industrial waste stream effluents
- PCB in transformer oil by Cl analysis
- Heavy metal air particulates collected on membrane filters, either from personnel samplers or from high volume samplers.
- Lead (Pb) in paint

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## 2.0 METHOD SUMMARY

The Spectrace 9000 Portable XRF Analyzer employs three radioactive isotope sources: iron-55 (Fe-55), cadmium-109 (Cd-109), and americium-241 (Am-241) for the production of primary X-rays. Each source emits a specific set of primary X-rays which excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the appropriate source is selected according to its excitation efficiency for the element of interest. See page 1-2 of the Spectrace 9000 Operating Instructions for a chart of source type versus element range.

The sample is positioned in front of the source-detector window and sample measurement is initiated which exposes the sample to primary radiation from the source. Fluorescent and backscattered X-rays from the sample enter through the beryllium (Be) detector window and are counted in the high resolution mercuric iodide (HgI<sub>2</sub>) detector.

Elemental concentrations are computed using a Fundamental Parameter (FP) algorithm of the form:

$$\text{Concentration} = R \times S \times (1 + \text{SUM}(A_i \times C_i))$$

"R" is the measured analyte X-ray intensity relative to the pure element; "S" is a calculated sensitivity coefficient. The quantity SUM{} is a summation of "n"-element absorption-enhancement terms containing calculated alpha-coefficients and iteratively computed element concentrations. The Spectrace 9000 utilizes FP XRF calibrations derived from theoretical considerations (as opposed to empirical data). The menu-driven software in the Spectrace 9000 supports multiple XRF calibrations called "applications." Each application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of FP calibration coefficients.

The measurement time of each source is user-selectable. The shorter source measurement times (15 -30s) are generally used for initial screening and hot spot delineation, while longer measurement times (30 - 500s) are typically used for higher precision and accuracy requirements.

## 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This SOP specifically describes equipment operating procedures for the Spectrace 9000; hence, this section is not applicable to this SOP.

## 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user or application related error. Generally, the instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally more significant and will vary with each site and method used. The components of the user or application related error are the following.

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#### 4.1 Sample Placement

This is a potential source of error because the X-ray signal decreases as the distance from the radioactive source is increased. However, this error is minimized by maintaining the same distance for each sample.

#### 4.2 Sample Representivity

In order to accurately characterize site conditions, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability. For additional information on representative sampling, refer to the "Removal Program Representative Sampling Guidance, Volume 1 - Soil."<sup>(1)</sup>

#### 4.3 Reference Analysis

Soil chemical and physical matrix effects may be corrected by using site-specific soil samples which have been analyzed by Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectroscopy as calibration samples. A major source of error can result if these samples are not representative of the site and/or if the analytical error is large. Additionally, when comparing XRF results with reference analyses results, the efficiency of the sample digestion reference analysis should be considered. Some digestion methods may breakdown different sample matrices more efficiently than others.

#### 4.4 Chemical Matrix Effects (Due to the Chemical Composition of the Sample)

Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, iron (Fe) tends to absorb copper (Cu) X-rays, reducing the intensity of Cu measured by the detector. This effect can be corrected mathematically through the use of FP coefficients.

#### 4.5 Physical Matrix Effects (Due to Sample Morphology)

Physical matrix effects are the result of variations in the physical character of the sample. They may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much coarser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles; this results in a higher intensity reading for that sample and.

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consequently, an apparently higher measured concentration for that element.

#### 4.6 Application Error

Generally, the error in the application calibration model is insignificant (relative to the other sources of error) PROVIDED the instrument's operating instructions are followed correctly. However, if the sample matrix varies significantly from the design of the application, the error may become significant (e.g., using the soils application to analyze a 50 percent iron mine tailing sample).

#### 4.7 Moisture Content

Sample moisture content will affect the analytical accuracy of soils or sludges. The overall error may be secondary when the moisture range is small (5-20 percent), or it may be a major source of error when measuring the surface of soils that are saturated with water.

#### 4.8 Cases of Severe X-ray Spectrum Overlaps

When present in the sample, certain X-ray lines from different elements can be very close in energy and, therefore, can interfere by producing a severely overlapped spectrum.

The typical spectral overlaps are caused by the  $K_{\alpha}$  line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with the  $K_{\alpha}$  line of element Z. This is the so-called  $K_{\alpha}/K_{\alpha}$  interference. Since the  $K_{\alpha}/K_{\alpha}$  intensity ratio for the given element usually varies from 5:1 to 7:1, the interfering element, Z-1, must be present in large concentrations in order to disturb the measurement of analyte Z. The presence of large concentrations of vanadium (V) could disturb the measurement of chromium (Cr). The V  $K_{\alpha}$  and  $K_{\beta}$  energies are 4.951 and 5.427 Kev, respectively. The Cr  $K_{\alpha}$  energy is 5.41 Kev. The resolution of the detector is approximately 270 eV. Therefore, large amounts of V in a sample will result in spectral overlap of the V  $K_{\beta}$  with the Cr  $K_{\alpha}$  peak (see Figure 1, Appendix A) and the measured X-ray spectrum will include TOTAL counts for Cr plus V lines.

Other interferences arise from K/L, K/M, and L/M line overlaps. While these are less common, the following are examples of severe overlap:

As  $K_{\alpha}$ /Pb  $L_{\alpha}$ , S  $K_{\alpha}$ /Pb  $M_{\alpha}$

In the arsenic (As)/lead case, Pb can be measured from the Pb  $L_{\alpha}$  line, and arsenic from either the As  $K_{\alpha}$  or the As  $K_{\beta}$  line; this way the unwanted interference can be corrected. However, due to the limits of mathematical corrections, measurement sensitivity is reduced. Generally, arsenic concentrations can not be efficiently calculated in samples with Pb:As ratios of 10:1 or more. This may result in zero arsenic being reported regardless of what the actual concentration is.

The Spectrace 9000 uses overlap factors to correct for X-ray spectral overlaps for the elements of interest for a given application.

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## 5.0 EQUIPMENT / APPARATUS

### 5.1 Description of the Spectrace 9000 System

The analyzer utilizes the method of Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometry to determine the elemental composition of soils, sludges, aqueous solutions, oils, and other waste materials.

The Spectrace 9000 analyzer includes three compact, sealed radiation sources contained in a measuring probe: Fe-55, Cd-109, and Am-241. The analyzer software automatically selects which sources to use as well as measurement time for each source based on stored information for each application. The probe is equipped with a high resolution HgI<sub>2</sub> detector, which is connected by cable to an environmentally sealed electronic module.

The electronic unit provides internal non volatile memory for storage of 120 spectra and 300 multi-element analysis reports. An RS-232 serial port is provided for downloading data and spectra to a peripheral device. The multi-element analysis reports and the 2000-channel spectra can be displayed on the instrument's LCD panel. The replaceable and rechargeable internal battery provides for field-portable operation.

The Spectrace 9000 is supplied with three factory-installed FP-based applications (calibrations). The "Soil Samples" application is for analysis of soils where the balance of the sample (that portion not directly measured by the instrument) is silica (SiO<sub>2</sub>). The "Thin Film" application is for analysis of thin films such as air monitoring filters or wipes. Finally, the "PbK in Paint" application is for analyzing Pb in paint films and is reasonably independent of the type of substrate. Spectrace Instruments will also develop calibrations to meet new user application requirements (e.g., adding elements to the present "Soil Samples" application). The PC-based Spectrace 9000 Application Generator software may also be used to develop new applications.

The Spectrace 9000 can be powered from a 115-volt (or 220-volt) wall outlet or from its 4-hour capacity battery. It can be operated in temperatures ranging from 32 to 120° Fahrenheit (F). Furthermore, the probe and electronic unit may be exposed to a light rain. However, additional protection is provided when the system (electronic unit and probe) is contained in the optional water repellant carrying case.

### 5.2 Equipment and Apparatus List

#### 5.2.1 Spectrace 9000 Analyzer System

The complete Spectrace 9000 Analyzer System includes:

- Analyzer unit for data acquisition, processing, and display
- Hand-held probe including:
  - High-resolution HgI<sub>2</sub> detector
  - Three excitation sources (<sup>55</sup>Fe, <sup>109</sup>Cd, <sup>241</sup>Am)

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- Safety cover
- Probe laboratory stand with the following:
  - Base for table top use
  - Safety shield over sample
  - Positioning fixtures for standard 30-mm and 40-mm X-ray sample cups
- Interconnecting cable
- RS-232C Serial I/O Interface cable
- Two blank check samples
- Pure element check samples
- Battery charger
- Battery pack
- System carrying/shipping case
- Spectrace 9000 Operating Instructions, application software, and utilities software. The application software is specific to each unit and cannot be interchanged between different units. The software is identified by the serial number of the unit.

#### 5.2.2 Optional Items

- 31-mm diameter sample cups
- XRF polypropylene film, 0.2 mil thick
- Field carrying case
- Peripheral devices such as a printer and IBM compatible Personal Computer (PC)
- Spare probe window assembly
- Spare battery pack, charger, and charger adaptor (required to charge spare battery outside of electronic unit)

See the Spectrace 9000 Accessories Price List for additional options.

For mobile lab or laboratory X-ray sample preparation accessories (such as drying ovens, grinders, sieves, etc.), consult general laboratory equipment suppliers.



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#### 5.2.3 Limits and Precautions

The probes should be handled in accordance with the following radiological control practices.

1. The probe should always be in contact with the surface of the material being analyzed, and that material should completely cover the probe opening (aperture) when the sources are exposed. Do not remove a sample or move the probe while the indicators show SOURCE ON.

SOURCE ON indicators are:

- the message on the screen "SOURCE ON"
  - the flashing light at the base of the probe.
2. When the sources are exposed, under no circumstances should the probe be pointed at the operator or surrounding personnel.
  3. Do not place any part of the operator's or co-worker's bodies in line of exposure when the sources are exposed or partially covered.
  4. The probe must be covered with the safety cover or laboratory safety shield when not in use.
  5. Spectrace Instruments must be notified immediately of any condition or concern relative to the probe's structural integrity, source shielding, source switching condition, or operability.
  6. The appropriate state agency or the Nuclear Regulatory Commission (NRC) office must be notified immediately of any damage to the radioactive source, or any loss or theft of the device (see factory supplied data on radiological safety).
  7. Labels or instructions on the probe(s) must not be altered or removed.
  8. The user must not attempt to open the probe.
  9. The source(s) in the probe must be leak-tested every 6 months as described in the Spectrace 9000 Operating Instructions. The leak test certificates must be kept on file, and a copy must accompany the instrument at all times.
  10. The probe laboratory safety shield assembly must be used when the probe is inverted for measuring samples contained in cups.

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11. During operation, the probe must be kept at least 10 feet from computer monitors and any other source of radio frequency (RF). Some monitors have very poor RF shielding and will affect measurement results.
12. The Spectrace 9000 should not be dropped or exposed to conditions of excessive shock or vibration.
13. The electronic unit should be left on whenever the battery charger is connected to it. If the electronic unit is shut off with the battery charger plugged in, the battery may be damaged due to overcharging.

#### Additional precautions include:

1. The probe cable must never be pulled while unplugging the probe. The probe plug should be grasped at the ribbed metal connector and squeezed and pulled gently while the connector is unplugged. The connector must never be forced when plugging in the connector.
2. The handle of the electronic unit must not be rotated unless the release buttons on each side of the handle are depressed.
3. The Spectrace 9000 should not be stored at an ambient temperature below -4°F or above 110°F.
4. The battery charging unit should only be used indoors in dry conditions.
5. Battery packs should be changed only in dry conditions.

#### 5.3 Peripheral Devices

The Spectrace 9000 may be used with a wide range of peripheral devices for electronic data capture or printed readout as long as they are compatible with the RS-232 serial I/O protocol. Such devices include terminals, printers, electronic data loggers, personal computers, etc.

##### 5.3.1 Communication Cable Connection

Plug the 25-pin connector of the RS-232 Serial I/O cable into the Spectrace 9000 25-pin connector (the connection just below the display screen on the electronic unit) and the 9-pin connector of the cable into the serial port of the receiving device.

##### 5.3.2 Communication Port Setup

To communicate with an external device, the Spectrace 9000 MUST be set at the same baud rate, word length, and parity as the receiving device. The Spectrace 9000 allows you to select various configurations for these parameters in the communication (Comm.) port setup portion of the More submenu (which can be accessed from the main menu).

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The default COM setup for application and utilities software is 9600.N.8.1.

#### 5.3.3 User Software

Refer to your PC software manual for details on additional settings that may be required for proper interfacing between the Spectrace 9000 and your particular software.

#### 5.4 Instrument Maintenance

##### 5.4.1 Probe Window

Should the probe window become damaged or punctured, it should be replaced as soon as possible to prevent dust and moisture from entering the probe. Replacement window assemblies can be ordered from Spectrace Instruments. Note the location of the window aperture: it is closer to one end of the window plate. Simply unscrew the old window plate, press any corner of it, and remove it. Stretch the O-ring for 10 seconds, and lay it back in the groove. The O-ring must lie flat in the groove in order for the new window plate to be installed. Install the new window assembly in the same manner as the old. If the surface of the window plate is not flush with the face of the probe, the O-ring has probably come out of the groove. Remove the assembly, and try the same procedure again.

##### 5.4.2 Further Information and Troubleshooting

Refer to the Spectrace 9000 Operating Instructions for additional detailed operational and/or maintenance and troubleshooting instructions. If no solution is found in the manual, contact Spectrace Instruments for assistance.

An instrument log should be maintained to document specific corrective actions taken to alleviate any instrumental problems, or for recording any service that has been performed.

#### 6.0 REAGENTS

Generally, calibration standards are not necessary for site screening and extent of contamination analyses with the Spectrace 9000. Optionally, an application (only the Soil Sample application will be discussed here) can be optimized or verified to be 1:1 proportional to another analytical (reference) method (see Section 9.3 and 10.1). This can be done by analyzing a suitable set of Site-Specific Calibration Standards (SSCS) or Standard Reference Materials (SRMs) and performing a regression analysis on the reference (dependent) and the Spectrace 9000 results (independent) for each element of concern. SSCS and SRMs must be representative of the sample matrix to be analyzed by XRF, for example, National Institute of Standards and Technology (NIST) SRMs 2709, 2710, and 2711 for the soil application. In an application, any element's calibration can be adjusted by entering the desired slope and offset (intercept) in the Adjust Calibration menu. If any element's calibration has been adjusted in an application, "adj" will appear on the results screen. An adjusted element calibration can always be changed back to the initial slope and offset values of 1 and 0, respectively.

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## 7.0 PROCEDURE

### 7.1 Prerequisites

If the Spectrace 9000 will be used in a location where AC power outlets are conveniently accessible, connect the battery charger to the electronic unit and plug the charger cord into the outlet. The probe cable must be connected before switching on the power. Plugging and unplugging this cable with the power on can damage the detector.

To connect the battery, set the electronics unit on its face and use a flat blade screwdriver to loosen the two one-quarter turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap covering the three-pronged plug. Remove the cap and plug it into the battery pack. Put the battery pack into the unit and tighten the fasteners.

Apply power to the Spectrace 9000 by pressing the <ON> button. The electronic unit may not come on with the battery charger hooked up if the battery has been totally drained. The drained battery may require a 10 minute charge prior to startup. In a few seconds the display shows the version of software. If necessary, adjust the contrast knob located on the underside of the front display. This knob can be turned so far that the display appears blank.

The initial screen displays for about 10 seconds and then a prompt will ask if the time and date are set correctly. The date **MUST** be set correctly otherwise serious errors in source-decay compensation can result. Additionally, results tables include the time and date of analysis. The main menu appears after the time and date screens.

If a "battery low" message appears, recharge or change the battery before proceeding, or operate the unit using line voltage.

Allow the Spectrace 9000 to warm up for approximately 30 minutes after it has been turned on before performing analysis.

#### 7.1.1 Gain Control

Automatic gain compensation is a feature of both Soil and Thin Samples applications, which allows operation of the instrument over a wide range of ambient temperatures and from one day to another without standardization. To maintain gain control compensation, it is necessary to occasionally operate with a minimum acquisition time of 50 seconds on the Cd-109 source. If the automatic gain control fails or is out of range, an error message will appear on the screen. If the error message continues to appear after repeat analyses, then the Cd-109 measurement time should be checked and/or an energy calibration should be performed. If the problem continues, contact Spectrace Instruments for help.

#### 7.1.2 Setting Data and Spectrum Store/Send Mode

The Set store/send modes option is located in the More screen which can be accessed from the main menu. Data and/or Spectrum storage must be enabled for automatic on-

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board storing to occur. Sufficient memory is available to store up to 300 sets of analysis results and up to 120 spectra (40 samples since each sample has three spectra). When the available memory is full, the respective spectra or results storage mode is automatically disabled. The spectra or results memory must be cleared (deleted) and the respective store mode enabled before results and/or spectra can be stored again.

## 7.2 General Keys and Menu Software

This section outlines the general keys and basic menu software. Flow charts which describe the menu structure in detail are located on pages 4-13 through 4-17 in the Spectrace 9000 Operating Instructions.

### 7.2.1 The Keyboard

The row of numeric keys under the LCD screen performs functions defined by labels (a menu) written to the bottom line of the display by the Spectrace 9000 software. As the operator moves through the various menus, the keys are redefined to provide an efficient user interface.

The keypad to the right of the screen is used for numeric entry. The <Cont/Pause> key (referred to as the <Cont>) is used:

- to enter information as an <Enter> key
- to begin an analysis
- to pause an analysis in progress

The left arrow <←> key is used to edit entries before pressing <Cont>.

### 7.2.2 The Measure (Ready) Screen

This main menu selection displays the application name, revision date, measurement time for each source, and accesses other options (see flow diagrams in Spectrace 9000 Operating Instructions).

### 7.2.3 The Choose an Application Screen

This main menu selection lists the applications currently loaded in the unit. Applications are selected and source measurement times may be modified in this screen (see flow diagrams in Spectrace 9000 Operating Instructions).

### 7.2.4 The Review Stored Results Screen

This main menu selection lists the stored results. *Up* and *Down* scroll are used on many screens. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. Stored results may be reviewed,

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deleted, or downloaded to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions).

#### 7.2.5 The Review Stored Spectra Screen

This main menu selection lists the stored spectra which may be deleted or transmitted to the COM port (see flow diagrams in Spectrace 9000 Operating Instructions). You cannot display spectra under this screen. Spectra may be displayed in the *Examine Spectrum* portion of the *More* screen (accessed from the main menu) or in the *Examine Spectrum* selection from the *Results* screen under the *More Options* menu selection.

#### 7.2.6 The More (Other Functions) Screen

This main menu selection lists the following functions:

- *Set clock/calendar*
- *Comm. port setup*
- *Set store/send modes*
- *Application maintenance*
- *Examine spectrum*

#### 7.2.7 The Results Screen

The Results screen is displayed at the end of the analysis. If the automatic *Store Results* mode is enabled, you will be prompted for sample identification (*ID*) before the Results screen is displayed. *Up* or *Down* scrolls the screen to view more results. When *Up* and *Down* are displayed, pressing the <0> (zero) key will toggle to *PgUP* and *PgDN* for rapid movement through long lists. *Send* transmits results to the COM port. *Store* prompts for an *ID* and then stores results in memory. *Measr* will immediately begin another analysis cycle. *Optr* displays the first of two screens listing special options under the Results screen (the second screen is located under *More Optr* of the first screen. See flow diagrams in Spectrace 9000 Operating Instructions). The most frequently used functions are the *Examine Spectrum* and *Enable/Disable Display Thresholds* located on the second screen of options.

### 7.3 Preoperational Checks

#### 7.3.1 Energy Calibration Check

An energy calibration should be performed after an instrument is shipped and periodically (approximately 2 weeks) to ensure proper energy calibration. The *Energy Calibration* function is located in the *Options* section of the *Measure* Screen. You will be prompted to place the safety shield on the probe and then initiate a 600- second analysis that will update the X-ray energy calibration.

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The energy calibration check is performed in the field daily and after an energy calibration to verify proper energy calibration. To perform an energy calibration check, place the safety shield on the probe. Select the *Soil Samples* application and measure the safety shield using a minimum acquisition time of 60 seconds for each source. Save the results and spectra for documentation. Select *Optis*, *More Options*, and then *Examine Spectrum*. Examine the spectrum of each source. Locate and record the centroid KeV (using the x12 horizontal magnification) for each of the following peaks:

Source	Peak	Theoretical (KeV)	Specification (KeV)
Cd-109	Pb L-alpha	10.54	± 0.040
	Pb L-beta	12.61	± 0.040
	Pb L-gamma	14.76	± 0.040
	Source line	22.10	± 0.040
Fe-55	S K-alpha	2.31	± 0.020
	Source line	5.89	± 0.020
Am-241	Pb L-alpha	10.54	± 0.050
	Pb L-beta	12.61	± 0.050
	Source line	59.5	± 0.200

Perform an *Energy calibration* (see Spectrace 9000 Operating Instructions) and then do another energy calibration check if any of the peaks fail to meet specification. The energy calibration check should be performed once at the beginning of the day, after an energy calibration, after loading an application, and whenever the instrument exhibits a persistent drift.

#### 7.3.2 Resolution Check *End*

The resolution check examines the detector's ability to resolve X-ray energies. This should be performed once at the beginning of the day. Select the *Soil Samples* application, and measure a sample of iron using a minimum acquisition time of 60 seconds for the Cd-109 source. Save the results and spectra for documentation. Select *Examine spectrum* under the More Options section of the Results screen. Examine the Cd-109 spectrum. Locate and record the maximum peak counts (must be >1000 counts) of the iron K-alpha peak (6.4 KeV) using the x12 horizontal magnification (see Figure 2, Appendix A). Divide the maximum peak counts by two. Examine the right (high energy) side of the peak and record the counts and KeV of the channel with counts less than or equal to one-half the maximum peak count value (channel B, Figure 2). Examine the left (low energy) side of the peak and record the counts and KeV of the channel with counts less than or equal to one-half the maximum peak count value (channel A, Figure 2). Subtract the left-side KeV from the right-side KeV (KeV at B - KeV at A, Figure 2). The difference should be less than 0.300 KeV. If the unit fails to meet this specification, call Spectrace Instruments for assistance.

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#### 7.3.3 Blank (Zero) Sample Check

The blank (Zero) sample check is performed to monitor the instrument's zero drift in the selected application. The blank sample check and the *Acquire Background Data* operation (discussed below) only apply to the application currently selected. This should be done once at the beginning of the day, after an energy calibration, after loading an application, and whenever the instrument exhibits a persistent drift on a blank or low-level sample.

Mount the probe in the laboratory stand and select the *Soil Samples* application. Disable the display thresholds. This will permit results less than one standard deviation (STD) to be displayed (even negatives). Measure the quartz blank provided with the unit (or a "clean" sand sample) using a minimum acquisition time of 60 seconds for each source. Review the results table. All elemental results for target elements with atomic number 24 (Cr) and higher in the periodic table should be within 3 standard deviations of zero ( $0 \pm 3 \cdot \text{ISTDI}$ ); all non-target element results should be within 5 standard deviations ( $0 \pm 5 \cdot \text{ISTDI}$ ). Repeat the measurement if the unit fails to meet these specifications. If several elements continue to be significantly out of these specifications, check the probe window and the blank sample for contamination or perform the *Acquire background data* operation located in the Measure (Ready) screen option. Perform the blank (Zero) sample check again. Save the results and spectra for documentation. Enable the display thresholds prior to sample analysis after the blank sample check procedure is completed.

#### 7.3.4 Target Element Response Check

The purpose of the target element response check is to ensure that the instrument and the selected application are working properly prior to performing sample analysis. This check should be performed at the beginning of the day. Use low, mid, and high samples, or standards with known concentrations for some or all of the target elements to be checked. Select a low sample near the quantitation limit of the target elements. Select a mid sample near the site action level and a high sample near the maximum concentration of the target elements expected on site.

These samples should be measured using the same source acquisition times that will be used for sample analysis. Save the sample check results and spectra for documentation.

#### 7.4 Selecting Source Measuring Time

The source measuring time may be modified under the Measure screen. Zero (seconds) measuring time should never be selected for any source for any application. Generally, the element detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Although counting statistics improve as measurement time increases, the practical limit for typical applications is 600 to 800 seconds. The elements are grouped together according to the radioisotope used for their excitation with typical minimum detection limits shown in Sections 7.4.2. and 7.4.3.



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Automatic gain compensation is a feature of both the Soil and Thin Samples applications which allows operation of the instrument over a wide range of ambient temperatures and from one day to another without standardization. To maintain this gain control compensation, it is necessary to occasionally operate with a minimum acquisition time of 50 seconds on the Cd-109 source.

The *Real/live* option toggles between real time (true clock time) and live time (total time the instrument is counting). The latter adds time to the analysis to correct for the time the system is busy processing pulses.

#### 7.4.1 Minimum Source Measuring Times

A minimum measuring time (real or live) of 15 seconds for the Fe-55 source, 30 seconds for the Cd-109 source, and 10 seconds for the Am-241 source is recommended when using the Soil Samples application. Measuring times for a source that excites a target element can be increased if lower detection limits are required.

When using the Thin Samples application, the measuring time for any source may be reduced to 10 seconds if the source does not excite a target element since this application does not correct for interelement effects. If a source excites a target element, a minimum measuring time (real or live) of 60 seconds for the Fe-55 source, 60 seconds for the Cd-109 source, and 120 seconds for the Am-241 source is recommended.

A minimum of 60 seconds is recommended for the Cd-109 source when using the PbK in Paint application.

#### 7.4.2 Typical Minimum Detection Limits (MDLs) for the Soil Samples Application

For source measuring times of 60 seconds, typical element MDLs (in milligram per kilogram, mg/kg) for the Soil Samples application are:

Source	Element	MDL (mg/kg)
Fe-55	Potassium (K)	325
	Calcium (Ca)	150
	Titanium (Ti)	110
	Chromium (CrLo)	180
Cd-109	Chromium (CrHi)	525
	Manganese (Mn)	410
	Iron (Fe)	225
	Cobalt (Co)	205
	Nickel (Ni)	125
	Copper (Cu)	90
	Zinc (Zn)	50
	Mercury (Hg)	60

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	Arsenic (As)	50
	Selenium (Se)	35
	Lead (Pb)	30
	Rubidium (Rb)	10
	Strontium (Sr)	10
	Zirconium (Zr)	10
	Molybdenum (Mo)	10
Am-241	Cadmium (Cd)	180
	Tin (Sn)	100
	Antimony (Sb)	65
	Barium (Ba)	20

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the matrix of the soil standard used to calculate MDLs, age of sources, moisture content, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Additionally, more elements may be added to the Soil Samples application. Contact Spectrace Instruments for information about modifications to applications.

#### 7.4.3 Typical Minimum Detection Limits (MDLs) for the Thin Samples Application

For source measuring times of 200 seconds for the Fe-55 and Cd-109 sources, and 800 seconds for the Am-241 source, typical element MDLs (in microgram per square centimeter,  $\mu\text{g}/\text{cm}^2$ ) for the Thin Samples application are:

Source	Element	MDL ( $\mu\text{g}/\text{cm}^2$ )
Fe-55	Potassium (K)	0.40
	Calcium (Ca)	0.20
	Titanium (Ti)	0.15
	Chromium (CrLo)	0.40
Cd-109	Chromium (CrHi)	0.90
	Manganese (Mn)	0.65
	Iron (Fe)	0.65
	Cobalt (Co)	0.50
	Nickel (Ni)	0.30
	Copper (Cu)	0.65
	Zinc (Zn)	0.40
	Mercury (Hg)	0.45
	Arsenic (As)	0.40
	Selenium (Se)	0.15
	Lead (Pb)	0.50
	Rubidium (Rb)	0.10

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	Strontium (Sr)	0.10
	Zirconium (Zr)	0.15
	Molybdenum (Mo)	0.10
Am-241	Cadmium (Cd)	2.5
	Tin (Sn)	2.5
	Antimony (Sb)	1.5
	Barium (Ba)	0.70

NOTE: These typical MDLs are provided as an aid for selecting source measurement times; observed values for a given situation may vary depending on the thin sample standard used to calculate MDLs, age of sources, and other factors discussed in Section 4.

Generally, the detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Use of thick filters or filters with high background or contamination will result in higher MDLs and require a background subtraction. Additionally, more elements may be added to the Thin Samples application. Contact Spectrace Instruments for information about modifications to applications.

## 7.5 Sample Handling and Presentation

When making XRF measurements, be sure to maintain constant measurement geometry in order to minimize variations in analysis results. Document any anomalies in measurement geometry, sample surface morphology, moisture content, sample grain size, and matrix (see Section 4.0).

### 7.5.1 Soil Samples

Soil samples may be analyzed either in-situ or in prepared X-ray sample cups. The Soil Samples application assumes the sample to be infinitely thick. For in-situ measurements this is almost always the case. However, for sample cup measurements it is advisable to fill the cup nearly full and tap it on the bench to compact the soil. This ensures that the sample is as uniformly thick as possible from analysis to analysis. The Spectrace 9000 laboratory stand and safety shield should be used when analyzing sample cups.

An area for in-situ analysis should be prepared by removing large rocks and debris. The soil surface should be rendered flat and compact prior to analysis. The Spectrace 9000 probe should be held firmly on the ground to maximize instrument contact with the ground. The probe should not be moved during analysis. Analysis of water saturated soils should be avoided. A thin layer of 0.2-mil polypropylene XRF film may be mounted on the surface probe to minimize contamination. Use of varying thicknesses of plastic (bags) have been shown to interfere in the light element (low atomic number) measurement and may affect the FP calibration of the other element concentrations.<sup>(2)</sup> Additionally, plastic may contain significant levels of target element contamination.

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Course-grained soil conditions or nuggets of contaminated material may preclude a truly representative sample and adversely affect the analysis results (typically by under reporting the target element). Such samples should be prepared before analysis. Preparation consistency is important to minimize variation in analytical results.

This application is specifically designed for soil with the assumption that the balance of the material is silica. If samples with a much lighter (lower atomic number) balance are analyzed, the results will typically be elevated by a factor of two to four. Contact Spectrace Instruments for help in analysis of different matrices.

#### 7.5.2 Thin (Filter) Samples

The Thin Samples application is for analysis of thin samples such as filters or wipes. The detection limits are affected by the thickness of the substrate. Best results are obtained on the thinnest substrates. Always use the probe safety cover when measuring thin samples. This is not only for user safety, but also ensures a controlled background environment and provides a reference signal for the automatic gain control. Probe safety covers should never be interchanged between instruments.

Filters and wipes should be prescreened before use to establish background and contamination levels. Care should be used to prevent zinc oxide contamination from disposable gloves. Small 37-mm filters can be mounted between two layers of 0.2-mil thick polypropylene XRF film on 40-mm XRF cups for analysis. Larger filters can be placed on the probe with a sheet of 0.2- mil thick polypropylene XRF film between the filter and probe to prevent the window from being contaminated. Then the probe safety cover may be placed over the filter prior to analysis. Filters should be presented loaded side down and wrinkle free.

#### 7.5.3 Lead in Paint

The area selected for analysis should be smooth, representative and free of surface dirt. The Spectrace 9000 probe should be held firmly on the surface to maximize instrument contact. The probe should not be moved during analysis.

When used for specimen application (e.g., on paint chips or nonbacked films) remember to use the probe safety cover. In the PbK Application, you should also position a thick neutral sample, such as the quartz disk (blank), behind the specimen before closing the safety lid. Otherwise, the PbK X-rays excited in the safety cover will be sensed by the detector. In this application, do not perform the *Acquire background data* option from the list of options under the Ready screen.

## 8.0 CALCULATIONS

The Spectrace 9000 is a direct readout instrument that does not require any calculations.

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### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

#### 9.1 Precision

The precision of the method is monitored by reading a low- or mid-target element concentration sample (or SSCS or SRMs selected as described in Sections 6.0) at the start and end of sample analysis and after approximately every tenth sample. (A daily total of seven measurements is recommended.) Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action. Therefore, selection of a sample with a target element concentration at or near the site action level or level of concern is recommended. The sample is analyzed by the instrument for the normal field analysis time, and the results are recorded. The standard deviation for each target element is calculated. The relative standard deviation (RSD) of the sample mean can be used to calculate precision. The RSD should be within  $\pm 20$  percent for the data to be considered adequately precise.<sup>(7)</sup>

##### 9.1.1 The Method Detection Limit (MDL) and Method Quantitation Limit (MQL)

The MDL and MQL may be calculated from the measurement of either a low or blank sample, (or a SSCS or SRMs selected as described in section 6.0), at the start and end of sample analysis, and after approximately every tenth sample (a daily total of seven measurements is recommended). Alternatively, the quartz blank or "clean" sand may be used if a blank soil or sediment sample is unavailable.

Disable the display thresholds. This will permit results less than one standard deviation (STD) to be displayed (even negatives). Measure the sample using the same application and measuring time used for the samples. Enable the display thresholds prior to analyzing the next sample.

The sample standard deviation of the mean for each target element is calculated. If the standard deviation has a fractional component, round up to the next whole number prior to calculating the MDL and MQL.

The definition of the MDL is three times the calculated standard deviation value.

The definition of the MQL is 10 times the calculated standard deviation value.

#### 9.2 Reporting Results

All raw XRF data should be reported including the individual results of multiple analyses of samples and sampling points. The average and concentration range of each multiple analysis should also be reported.

A "reported" value for each analysis or average of multiple analyses should be processed in the following manner.

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1. Round the value to the same degree of significance contained in the SSCS or SRM sample assay values (usually two) if the element's calibration has been adjusted (see Section 6.0). Round to 2 significant figures for sample results. DO NOT round results for standards used to determine MDL or RSD values (use raw data).
2. Report all values less than the MMDL as not detected (ND).
3. Flag and note all values greater than or equal to the MMDL and less than the MQL (usually with a "J" next to the reported value).
4. Report all values equal to or greater than the MQL and within the linear calibration range (if the element's calibration has been adjusted [see section 6.0]).
5. Flag and note all values above the linear calibration range (greater than the highest SSCS used in the calibration adjustment procedure) if SSCS were used and the calibration was adjusted.

#### 9.3 Accuracy

Accuracy, relative to a specific digestion method and elemental analysis procedure, is determined by submitting an XRF analyzed sample (prepared sample cups may be submitted) for AA or ICP analysis at a laboratory.

The on-site analysis of soils by XRF instrumentation should be considered a screening effort only (QA1 data). Data derived from the instrument should be used with discretion. Confirmatory analyses on a subset of the screening samples (minimum 10 percent) can be used to determine if the XRF data meets QA2 data objectives. The confirmation samples should ideally be selected randomly from the sample set and include a number of samples at or near the critical level. The results of the metal analysis (dependent) and the XRF analysis (independent) are evaluated with a regression analysis. The correlation factor ( $R^2$ ) should be 0.7 or greater.<sup>(3)</sup>

XRF results may be multiplied by the slope prior to substitution for metal analysis results in contouring, kriging programs, or removal volume estimates. Correcting the XRF results based on confirmatory analyses should only be undertaken after careful consideration. It must be understood that the confirmatory analysis (AA or ICP) is an estimate of the concentration of metal contamination and is dependent upon the specific instrumentation and sampling methodology used. Since XRF is a total elemental technique, any comparison with referee results must account for the possibility of variable extraction, dependent upon the digestion method used and its ability to dissolve the waste or mineral form in question.

##### 9.3.1 Matrix Considerations

Other types of QA/QC verification should include verification that the instrument calibration is appropriate for the specific ~~matrix~~ to be assessed. This includes verification of potential multiple soil matrix types that may exist at a site. Matrix differences which affect the XRF measurement include large variations in calcium content, which may be

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encountered when going from siliceous to calcareous soils, as well as large variations in iron content.

### 10.0 DATA VALIDATION

#### 10.1 Confirmation Samples

Confirmation samples are recommended at a minimum rate of 10 percent and are required if QA2 data objectives have been established for site activities.<sup>(1)</sup> Ideally, the sample cup that was analyzed by XRF should be the same sample that is submitted for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a 6-inch by 6-inch area for both an XRF measurement and confirmation analysis.

The XRF and metals results are analyzed with a regression analysis using a statistical program such as SAS® or Stargraphics® with the intercept calculated in the regression. The correlation factor between XRF and AA/ICP data must be 0.7 or greater for QA2 data objectives.<sup>(1)</sup>

#### 10.2 Recording Results

Record all results and monitoring activities in a laboratory or field notebook. Alternatively, record results electronically on a hard drive or floppy disk.

#### 10.3 Downloading Stored Results and Spectra

Results (analytical reports) and spectra which have been stored in the Spectrace 9000 internal memory should be downloaded and captured in disk files on a PC (see section 5). Spectrace Instruments provides software for this purpose. Additionally, they provide software to prepare results or spectra for importing into a spreadsheet. Refer to the instructions provided with the programs for details on their operation.

Alternatively, other software with terminal data logging capabilities may be used to capture results and spectra to disk files.

After capturing results to a file, print a copy and save both the disk files and the printout for future reference and documentation purposes.

### 11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, corporate and/or any other applicable health and safety practices.

### 12.0 REFERENCES

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10. "Data Quality Objectives for Remedial Response Activities," EPA/540/G-87/004, March 1987.



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11. Rhodes, J., Stout, J., Schlinder, J., and Fiorek, S., "Portable X-ray Survey Meters for *In-Situ* Trace Element Monitoring of Air Particulates," American Society for Testing and Materials, Special Technical Publication 786, 1982, pp. 70 - 82.
12. Piorek, S., Rhodes, J., "In-Situ Analysis of Waste Water Using Portable Pre-concentration Techniques and a Portable XRF Analyzer," Presented at the Electron Microscopy and X-ray Applications to Environmental and Occupational Health Analysis Symposium, Penn. State Univ., Oct. 14 - 17, 1980.
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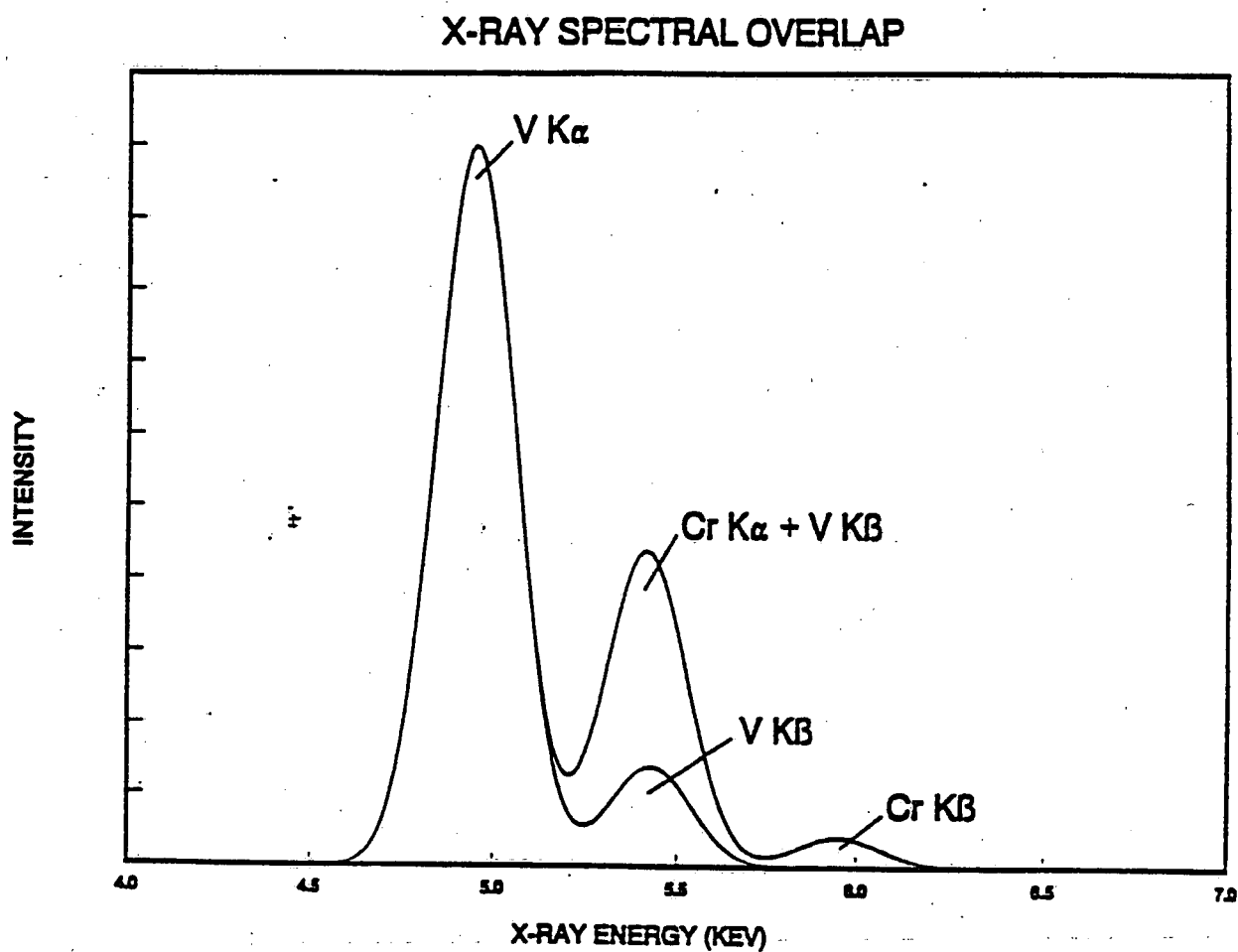
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FIGURE 1. X-Ray Spectral Plot Showing Overlap of Vanadium  $K_{\alpha}$  X-Rays in the Chromium  $K_{\alpha}$  Measurement Region.



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FIGURE 2. Iron X-Ray Spectrum Illustrating Detector Resolution Measurement

